[Second Edition.]

PATENT SPECIFICATION

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PROVISIONAL SPECIFICATION.

Improvements in and relating to the Surface Treatment of Aluminium and Aluminium Alloys.

We, ALFRED NORMAN DOUGLAS PULLEN, a British Subject, and THE BRITISH ALUMINIUM COMPANY, LIMITED, a company registered under the laws of Great Britain, both of the Company's address, Adelaide House, King William Street, London, E.C.4, do hereby declare the nature of this invention to be as follows:—

This invention relates to the surface treatment of aluminium and aluminium alloys and more particularly to treatment directed to the improvement of the specular reflectivity of such surfaces.

Sheet aluminium as produced by the normal rolling process has an average specular reflectivity for light of about 65%.

The reflectivity may be increased by processes such as polishing up to a figure of 75% to 80% but on exposure to air this enhanced reflecting power decreases rapidly until a figure equivalent to that for normal rolled sheet is reached.

The object of the present invention is the provision of a method and means by which the specular reflectivity of an aluminium or aluminium alloy surface may be increased very considerably in a cheap, efficient and permanent manner.

With this object in view, the present invention consists in a process for increasing the specular reflectivity of the surface of an aluminium or aluminium alloy article which comprises immersing the previously degreased article in an aqueous solution of sodium carbonate and sodium tri-phosphate to remove the normal oxide film, then in the same solution subjecting the article to anodic treatment by direct current at a moderately high current density for a short time to stop the attack of the solution upon the article and continuing the anodic treatment by direct current at a lower current density, and, after washing in water subjecting the article to further electrolytic treatment in a strong aqueous solution of sodium bisulphate.

The invention may be carried into effect in one way by way of example as follows:—

A piece of commercially pure rolled aluminium sheet is first degreased by suitable means such as cleaning in petrol, then, if necessary, it is buffed or otherwise polished mechanically. It is then given a chemico-electrical treatment in a bath having the following composition and under the following conditions.

ELECTROLYTE.

15% by weight of sodium carbonate, 5% by weight of trisodium phosphate in water. The solution to be heated to 80° C.±5°.

PROCEDURE.

The metal article is immersed in the bath with the result that solution of the metal by the alkaline electrolyte commences almost immediately. As soon as uniform attack is observed direct current at 5—6 volts is switched on, the aluminium being the anode, the current density applied being of the order of 30 amperes per square foot. An iron or steel cathode is used and procedure in the bath follows electro plating practice to some extent, e.g., the cathode is made to conform roughly to the anodic surface.

It is of advantage to have a bath which is of considerably larger dimensions than the electrodes. At the end of a short period, about 30 seconds, the current density automatically falls to about half the first value whilst the voltage is maintained approximately at the figure mentioned. The treatment is continued under these conditions for about 10 minutes. The article is then removed from the bath with the current still on but reduced to a small value by suitable means, and immediately washed in clean water. The surface of the metal, particularly that one facing the cathode, has now a very bright appearance and it will be found that if the original specular reflectivity value was, say 65%, it has now been raised to perhaps 80%. Besides the general bright appearance, it will be noticed that the surface of the metal now carries a thin film, presumably of aluminium oxide which shows brilliant interference colours.

For the following part of the treatment, that is to say, the provision of a protective oxide film, the metal is again

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subjected to an anodic process in another electrolyte, the composition of which and the conditions of operation being as follows.

ELECTROLYTE.

Aqueous solution containing 25 per cent. by weight of sodium bisulphate.

VOLTAGE.

6—12 volts direct current, the aluminium being the anode.

TEMPERATURE OF ELECTROLYTE.

30°—40° C.

TIME OF TREATMENT.

10—15 minutes.

The current density is of the order of 5 amperes per square foot.

GENERAL.

The figures given in the foregoing example are capable of modification without departing from the scope of the invention.

For example, for the first part of the treatment the composition of the electrolyte may include all strong alkaline electrolytes having a *p*H value of over 10 with alkalinities due to the hydroxides or carbonates of the alkali metals such as sodium potassium, lithium, with or without the addition of alkali metal salts such as, for instance, phosphates, borates or chromates.

The temperature range of the electrolyte may extend from about 40° to about 100° C.

The applied voltage may be varied between limits of about 2 to about 20 volts. For this part direct current is to be used, which term is intended to include unidirectional current of any kind.

For the second part of the treatment the electrolyte may comprise, in addition to the aqueous solution of sodium bisulphate, aqueous solutions of such substances as sulphuric acid, chromium sulphate, chromium oxalate, chromic acid, boric acid, phosphoric acid, acetic acid or salts of such acids. The percentage of sodium bisulphate may be from 5 to 40% by weight, the temperature may be from 20—50° C. and the voltage from about 5 to about 30 volts. For this part the current may be of a direct or alternating character.

The film, presumably of aluminium oxide, produced by the second part of the process hereinbefore described, viz., the protective coating for the brightened surface is relatively hard but is quite transparent and is to be distinguished from similar oxide films formed in other electrolytes in that it does not reduce the reflectivity value of the film produced in the first part of the process, in fact, in many instances it has been found that the reflectivity value has been slightly raised.

The process according to the invention is applicable to all kinds of surfaces either normal, polished or mat and in all cases results in a considerable increase of the specular reflectivity of the original material.

Whilst the foregoing particular example deals only with commercially pure rolled aluminium sheet, the scope of the invention is not limited thereto and aluminium alloys are included in the materials which are susceptible to beneficial treatment by the process according to the invention.

Dated this 15th day of December, 1934.

MARKS & CLERK.

COMPLETE SPECIFICATION.

Improvements in and relating to the Surface Treatment of Aluminium and Aluminium Alloys.

We, ALFRED NORMAN DOUGLAS PULLEN, a British Subject, and THE BRITISH ALUMINIUM COMPANY, LIMITED, a company registered under the laws of Great Britain, both of the Company's address, Adelaide House, King William Street, London, E.C.4, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to the surface treatment of aluminium and aluminium alloys and more particularly to treatment directed to the improvement of the specular reflectivity of such surfaces.

Sheet aluminium as produced by the normal rolling process has an average specular reflectivity for light of about 65 per cent.

The reflectivity may be increased by processes such as polishing up to a figure of 75 per cent. to 80 per cent. but on exposure to air this enhanced reflecting power decreases rapidly until a figure equivalent to that for normal rolled sheet is reached.

The object of the present invention is the provision of a method and means by which the specular reflectivity of an aluminium or aluminium alloy surface may be increased very considerably in a

cheap, efficient and permanent manner.

The present invention consists in a process for improving the reflectivity of a clean aluminium or aluminium alloy surface which comprises immersing the surface for a short time in an aqueous solution of sodium carbonate and sodium orthophosphate in the proportions of about 3 parts by weight of the carbonate to about 1 part by weight of the phosphate and having a *p*H value of 10 or more and a temperature of about 75° to 85° C. and then without removing the surface from the solution applying direct current anodic treatment thereto.

The invention further consists in a process as set forth in the preceding paragraph, including the further steps of washing the surface and providing a protective coating therefor.

The invention further consists in a process as set forth in the preceding paragraph wherein the protective coating is provided by direct or alternating current anodic treatment in an aqueous solution of sodium bisulphate ($NaHSO_4$).

The invention further consists in a process as set forth in the preceding paragraph wherein the sodium bisulphate solution contains from 5% to 40% by weight of the salt, is used at temperatures ranging from 20° C. to 50° C., and the current which may be direct or alternating in character is applied for about 10 to 15 minutes at a density of the order of 5 amperes per square foot of bright surface.

The invention further consists in a process as set forth in the preceding paragraph in which the time of immersion in the alkaline electrolyte is of the order of 10 seconds and the direct current anodic treatment is applied for about 10 to 15 minutes at a constant voltage which results in an initial current density of about 30 amperes per square foot of anode surface.

The invention further consists in the processes for treating the surface of an aluminium or aluminium alloy article substantially as hereinafter described.

The invention further consists in an aluminium or aluminium alloy article, the surface of which has been treated by the process or processes hereinafter described.

The invention may be carried into effect in one way by way of example as follows:—

A piece of commercially pure rolled aluminium sheet is first degreased by suitable means such as cleaning in petrol, then, if necessary, it is buffed or otherwise polished mechanically. It is then given a chemico-electrical treatment in a bath having the following composition and under the following conditions.

ELECTROLYTE.

15 per cent. by weight of sodium carbonate, 5 per cent. by weight of sodium ortho-phosphate in water. The solution to be heated to 80° C.±5°.

PROCEDURE.

The metal article is immersed in the bath with the result that the normal oxide film present on the metal is dissolved and solution of the metal by the alkaline electrolyte commences almost immediately. As soon as uniform attack is observed, approximately after 10 seconds immersion, direct current at 5—6 volts is switched on, the aluminium being the anode, the current density applied being of the order of 30 amperes per square foot. An iron or steel cathode is used and procedure in the bath follows electro-plating practice to some extent, e.g., the cathode is made to conform roughly to the anodic surface.

It is of advantage to have a bath which is of considerably larger dimensions than the electrodes. At the end of a short period, about 30 seconds, the current density automatically falls to about half the first value whilst the voltage is maintained approximately at the figure mentioned. The treatment is continued under these conditions for about 10 minutes. The article is then removed from the bath with the current still on but preferably reduced to a small value by suitable means, and immediately washed in clean water. The surface of the metal, particularly that one facing the cathode, has now a very bright appearance and it will be found that if the original specular reflectivity value was, say, 65 per cent., it has now been raised to perhaps 80 per cent. Besides the general bright appearance, it will be noticed that the surface of the metal now carries a thin film, presumably of aluminium oxide, which shows brilliant interference colours.

For the following part of the treatment, that is to say, the provision of a protective oxide film, the metal is again subjected to an anodic process in another electrolyte, the composition of which and the conditions of operation being as follows:—

ELECTROLYTE.

Aqueous solution containing 25 per cent. by weight of sodium bisulphate.

VOLTAGE.

6—12 volts direct current, the aluminium being the anode.

TEMPERATURE OF ELECTROLYTE.

30°—40° C.

TIME OF TREATMENT.

10—15 minutes.

The current density is of the order of 5 amperes per square foot.

Whilst a dual process has been described in the foregoing example, it is possible to carry out the process in two separate steps, a brightening and a final protective step which latter may, if necessary, be carried out by suitable known anodic processes.

GENERAL.

For the first part of the treatment the applied voltage may be varied between limits of about 2 to about 20 volts, and for this part direct current is to be used, which term is intended to include unidirectional current of any kind.

For the second part of the treatment the electrolyte may comprise, in admixture with the aqueous solution of sodium bisulphate, aqueous solutions of such substances as sulphuric acid, chromium sulphate, chromium oxalate, chromic acid, boric acid, phosphoric acid, acetic acid or salts of such acids. The percentage of sodium bisulphate may be from 5 to 40 per cent. by weight, the temperature may be from 20—50° C. and the voltage from about 5 to about 30 volts. For this part the current may be of a direct or alternating character.

The film, presumably of aluminium oxide, produced by the second part of the process hereinbefore described, viz. the protective coating for the brightened surface is relatively hard but is quite transparent and differs from similar oxide films formed in other, e.g. sulphuric acid or oxalic acid, electrolytes in that it does not reduce the reflectivity value of the film produced in the first part of the process, in fact, in many instances it has been found that the reflectivity value has been slightly raised.

The process according to the invention is applicable to all kinds of surfaces either normal, polished or mat and in all cases results in a considerable increase of the specular reflectivity of the original material.

Whilst the foregoing particular example deals only with commercially pure rolled aluminium sheet, the scope of the invention is not limited thereto and aluminium alloys are included in the materials which are susceptible to beneficial treatment by the process according to the invention.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for improving the reflectivity of the surface of a clean aluminium or aluminium alloy surface which comprises immersing the surface for a short time in an aqueous solution of sodium carbonate and sodium orthophosphate in the proportions of about 3 parts by weight of the carbonate to about 1 part by weight of the phosphate and having a pH value of 10 or more and a temperature of about 75° to 85° C. and then without removing the surface from the solution applying direct current anodic treatment thereto.

2. A process as claimed in Claim 1, including the further steps of washing the surface and providing a protective coating therefor.

3. A process as claimed in Claim 2 wherein the protective coating is provided by direct or alternating current anodic treatment in an aqueous solution of sodium bisulphate ($NaHSO_4$).

4. A process as claimed in Claim 3 wherein the sodium bisulphate solution contains from 5% to 40% by weight of the salt, is used at temperatures ranging from 20° C. to 50° C., and the current which may be direct or alternating in character is applied for about 10 to 15 minutes at a density of the order of 5 amperes per square foot of bright surface.

5. A process as claimed in Claim 4 in which the time of immersion in the alkaline electrolyte is of the order of 10 seconds and the direct current anodic treatment is applied for about 10 to 15 minutes at a constant voltage which results in an initial current density of about 30 amperes per square foot of anode surface.

6. The processes for treating the surface of an aluminium or aluminium alloy article substantially as hereinbefore described.

7. An aluminium or aluminium alloy article, the surface of which has been treated by the process or processes hereinbefore described.

Dated this 21st day of November, 1935.

MARKS & CLERK.

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